

Systematic Characterization of Upper Critical Fields for MgB₂ Thin Films by means of the Two-Band Superconducting Theory

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We present experimental results of the upper critical fields H_{c2} of various MgB₂ thin films prepared by the molecular beam epitaxy, multiple-targets sputtering, and co-evaporation deposition apparatus. Experimental data of the $H_{c2}(T)$ are successfully analyzed by applying the Gurevich theory of dirty two-band superconductivity in the case of $D_{\pi}/D_{\sigma} > 1$, where D_{π} and D_{σ} are the intraband electron diffusivities for π and σ bands, respectively. We find that the parameters obtained from the analysis are strongly correlated to the superconducting transition temperature T_c of the films. We also discuss the anomalous narrowing of the transition width at intermediate temperatures confirmed by the magnetoresistance measurements.

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I. INTRODUCTION

Recently, multiband superconductivity has attracted much attention since a novel metallic superconductor MgB₂ discovered at the beginning of the 21st century¹ has been revealed not only to show the highest T_c among intermetallic compounds but also to be a prototype two-band superconductor by numerous experimental and theoretical studies^{2,3,4,5,6,7,8,9}. The superconductivity of MgB₂ occurs in both σ and π bands of the hexagonal B layer which has a similar electronic structure to a graphite^{2,3,10,11}. The σ band, contributing to a chemical bond between B atoms, is two-dimensional orbital localized in the ab -plane of the hexagonal structure. On the other hand, the π band is an antibonding orbital along the c -axis, which spread out three-dimensionally. Therefore, the interaction between σ and π bands is very weak and then two superconducting gaps open below the superconducting transition temperature T_c in both bands.

Two-band superconducting character in MgB₂ appears in temperature dependence of the upper critical field H_{c2} , which shows an upward curvature below T_c . Superconducting anisotropy parameter γ determined from the ratio of H_{c2} for the ab -direction to that for the c -direction shows also temperature dependence. The γ value for the single crystal increases with decreasing temperature¹². Drastic enhancement of the H_{c2} is observed in dirty

samples such as the nonmagnetic impurity-doped MgB₂ bulks and/or films, where γ becomes small with showing a variety of the temperature dependence^{7,8,13,14,15}: in a case of a high resistivity film, the γ reduces with decreasing temperature⁷. These behaviors are not explained by only the dimensional crossover as usually discussed in the layered superconductors¹⁶, but the superimpose of the H_{c2} with two distinct superconducting gap^{5,9}.

There are mainly two disorder effects due to nonmagnetic impurity and/or crystal imperfection on the superconducting properties: one is suppression of T_c by weakening the superconducting coupling and the other is enhancement of the initial slope of the H_{c2} at T_c by reducing the electron mean free path¹⁷. In the two-band superconductor, there is very clever situation in which the great intraband scattering raises the H_{c2} and the small interband scattering affects little suppression on T_c . The shape of the $H_{c2}(T)$ curve is changed depending on which intraband scattering in the σ or π band is large. Gurevich gave the calculation formula for $H_{c2}(T)$ curves in the framework of dirty two-band superconductivity^{6,9}. He calculated the $H_{c2}(T)$ curves by using the parameters of the interband scattering g and the diffusivities D_{σ} and D_{π} in the σ and π bands including the anisotropy⁹. These parameters are good milestones in summarizing the experimentally obtained $H_{c2}(T)$ curves and in discussing the two-band superconductivity.

In this work, we present experimental and calculated results of the $H_{c2}(T)$ phase diagram of various MgB_2 thin films on some different substrates prepared by the molecular beam epitaxy (MBE), the multiple-targets sputtering, and co-evaporating deposition apparatus. We measured magnetoresistance in the whole region of $H_{c2}(T)$ curves by using a pulsed magnet up to 37 T. In order to obtain the best fitting parameters of the Gurevich theory to experimental data, we newly took a differential plot, $(-dH_{c2}/dT)$ vs. T , because this plot emphasizes the temperature dependence of the $H_{c2}(T)$ curve. We find that the parameters obtained from the analysis are strongly correlated to the T_c of the films. Moreover, we present another interesting result on the transition width. In the process of magnetoresistance measurements at several temperatures, we found that the resistive transition width does not monotonically increase with decreasing temperature but has a minimum at a certain temperature below T_c . We discuss the peculiar behavior in the temperature dependence of the transition width obtained from magnetoresistance measurements.

II. EXPERIMENT

H_{c2} was determined from measuring the magnetoresistance by dc four-terminal method at several temperatures below T_c down to 1.5 K under a pulsed magnetic field up to 37 T using a home-made pulsed magnet system. Magnetoresistance was measured in the field direction of both $H \parallel ab$ -plane and $H \parallel c$ -axis to obtain superconducting anisotropy of the films. A block diagram for the H_{c2} measurements is shown in Fig. 1. In order to avoid excessive electric noise and heat-inflow to the sample, a battery is used as a current source in a proper electric circuit. Currents of 1-40 mA are applied to MgB_2 films, depending on the resistance of the samples, to obtain about 3 mV signal from the normal resistance. The magnetic field is applied by triggering a thyristor switch of LCR circuit from a delay pulse generator. The pulsed width of magnetic field is 10 ms. A field trace is obtained by integrating the voltage data recorded in a digital oscilloscope from a field pick-up coil. A stray signal from the lead wires is cancelled by using a compensation coil and a bridge circuit.

Six as-grown MgB_2 thin films were used in the magnetoresistance measurements. Four MgB_2 films were prepared by Iwate group using an MBE apparatus with the co-evaporation conditions of low deposition rate in ultra-high vacuum without post annealing process^{18,19,20}. Two of them were deposited on MgO (100) (#1) and Si (111) (#2) substrates at 200 °C, the others were deposited on Ti buffer layers on ZnO (0001) substrates at 200 °C. The difference of the latter two films is the thickness of the Ti buffer layers; one is 10 nm (#3) and the other is 50 nm (#4). The film thickness and the resistivity at 40 K are listed in Table I. The structure and crystallinity were checked by x-ray diffraction. It was revealed that the c -

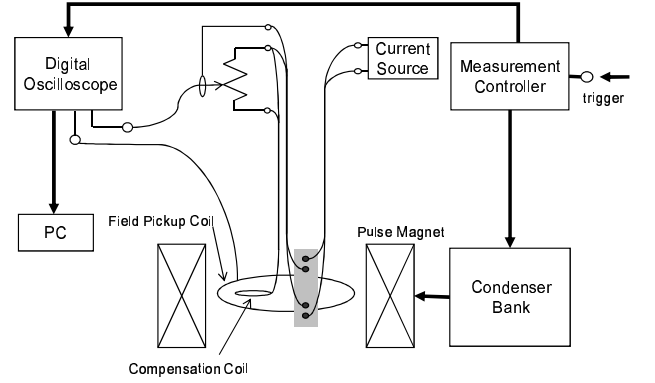


FIG. 1: Block diagram for the H_{c2} measurements under a pulsed magnetic field.

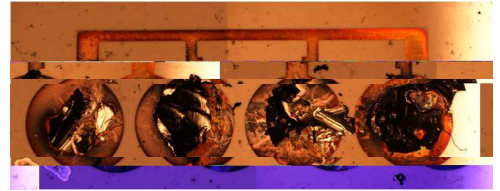


FIG. 2: Photograph of MgB_2 thin film #6 used in the H_{c2} measurements. The film width is 0.2 mm and the length between terminals is 2 mm.

axis of the MgB_2 orients a perpendicular direction to the film surface. Especially, the film #4 is found to have an excellent alignment with the in-plane orientation²⁰.

The other two MgB_2 films were prepared by NICT group. They were deposited on the c -plane of a sapphire substrate: one is fabricated by using a carousel-type multiple-targets sputtering system without any buffer layers²¹ denoted as #5, and the other is by using a co-evaporation method (#6)²². They have a smooth surface as grown at low-substrate temperatures and their c -axis also well orients a perpendicular direction to the film surface. The film thickness and the resistivity at 40 K are also listed in Table I. The co-evaporation film #6 was fabricated to the shape of a four-terminal pattern as shown in Fig. 2.

TABLE I: Fabrication conditions of MgB_2 thin films.

No.	thickness (nm)	$\rho(40\text{K})$ ($\mu\Omega\text{cm}$)	substrates	apparatus
#1	100	9.2	$\text{MgO}(100)$	MBE
#2	300	35	$\text{Si}(111)$	MBE
#3	200	40	$\text{ZnO}(0001)+10\text{nm Ti}$	MBE
#4	200	4	$\text{ZnO}(0001)+50\text{nm Ti}$	MBE
#5	160	270	$c\text{-Al}_2\text{O}_3$	Sputter
#6	700	44	$c\text{-Al}_2\text{O}_3$	Co-evaporation

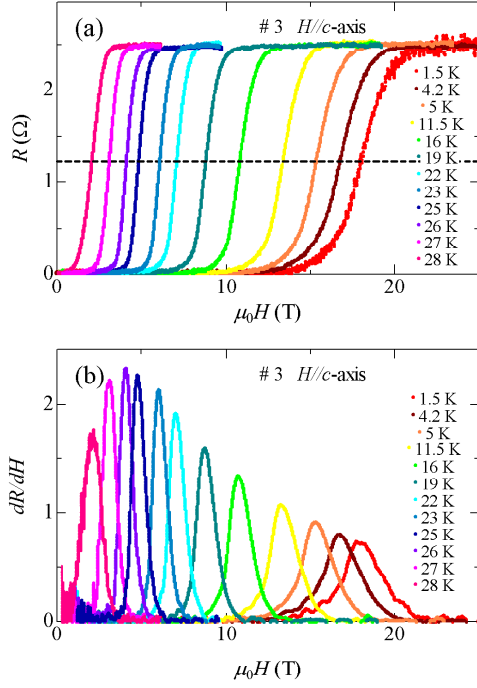


FIG. 3: (a) Magnetoresistance curves of MgB₂ film #3 for $H \parallel c$ -axis at several temperatures. (b) Differential plot of (a).

III. RESULTS

The magnetoresistance curves of the MgB₂ film #3 at several temperatures for $H \parallel c$ -axis are shown in Fig. 3 (a) as a typical example of the experimental data. No hysteresis is observed. The H_{c2} 's were defined as the midpoint of the resistive transition and the transition widths were defined as the fields between 10% and 90% of the normal resistance. The differential data dR/dH are plotted as a function of the magnetic field in Fig. 3 (b), where the peak fields coincide with the H_{c2} 's at each temperature. The peak width, which are almost proportional to the inverse of the peak heights, is also corresponding to the transition width. So, a maximum in the peak height observed at 26 K means that the transition becomes sharp at that temperature.

From the magnetoresistance measurements, we obtained the $H_{c2}(T)$ phase diagrams of six films, which are shown in Fig. 4. Error bars in the figure correspond to the fields between 10% and 90% of the normal resistance. Superconducting anisotropy parameter $\gamma_H = H_{c2}^{(ab)}/H_{c2}^{(c)}$ as a function of temperature was obtained as shown in the inset of the figure, where $H_{c2}^{(ab)}$ and $H_{c2}^{(c)}$ are the H_{c2} for $H \parallel ab$ -plane and $H \parallel c$ -axis, respectively. The $\mu_0 H_{c2}^{(ab)}(0)$'s attain to 20~40 T, which are apparently larger than those for MgB₂ single crystal. This indicates that the superconducting coherence length is effectively reduced through the reduction of the electron mean free path due to some kind of disorder in fabricating the films.

As for the anisotropy, the values of γ_H are between one and three depending on the films, which are smaller than that of single crystal MgB₂. γ_H decreases with increasing temperature, as shown in the inset of the figure. This is a basis for determining the ratio of the diffusivities for σ and π band as analyzed later. These experimentally obtained results of T_c , $\mu_0 H_{c2}^{(ab)}(0)$, $\mu_0 H_{c2}^{(c)}(0)$ and $\gamma_H(0)$ are listed in Table II.

IV. ANALYSIS

Experimentally obtained data of the $H_{c2}(T)$ and γ_H were calculated by fitting the Gurevich theory^{6,8,9} based on two-gap Usadel equations, in which impurity scattering is introduced as the intraband electron diffusivities and the interband scattering. The calculations were performed using an application software (Mathematica, Wolfram Research Co.). For two-gap superconductivity, T_c is not affected by intraband scattering, but decreased with increasing the interband scattering parameter g . We assume $T_{c0} = T_c(g = 0) = 40$ K and then determine the value of g from the Gurevich theory⁹ as follows,

$$U\left(\frac{g}{t_c}\right) + \frac{(\lambda_0 + w \ln t_c) \ln t_c}{p + w \ln t_c} = 0, \quad (1)$$

$$U(x) = \psi(1/2 + x) - \psi(1/2), \quad (2)$$

$$2p = \lambda_0 + [\lambda_- \gamma_- - 2\lambda_{\pi\sigma} \gamma_{\sigma\pi} - 2\lambda_{\sigma\pi} \gamma_{\pi\sigma}]/\gamma_+, \quad (3)$$

$$\gamma_{\pm} = \gamma_{\sigma\pi} \pm \gamma_{\pi\sigma}, \quad (4)$$

$$\lambda_{\pm} = \lambda_{\sigma\sigma} \pm \lambda_{\pi\pi}, \quad (5)$$

$$\lambda_0 = (\lambda_{\pm}^2 + 4\lambda_{\sigma\pi} \lambda_{\pi\sigma})^{1/2}, \quad (6)$$

$$w = \lambda_{\sigma\sigma} \lambda_{\pi\pi} - \lambda_{\sigma\pi} \lambda_{\pi\sigma}, \quad (7)$$

$$g = \hbar \gamma_+ / 2\pi k_B T_{c0}, \quad (8)$$

$$t_c = T_c / T_{c0}, \quad (9)$$

where $\psi(x)$ is a digamma function. $\lambda_{\sigma\sigma}$ and $\lambda_{\pi\pi}$ are the intraband superconducting coupling constants of the σ and π bands, respectively. $\lambda_{\sigma\pi}$ and $\lambda_{\pi\sigma}$ are the interband superconducting coupling constants and $\gamma_{\sigma\pi}$ and $\gamma_{\pi\sigma}$ are the interband scattering rates. There is a constraint among these interband parameters so as to satisfy the symmetry relation to the partial densities of states N_{σ} and N_{π} in the σ and π band, respectively:

$$\frac{\lambda_{\sigma\pi}}{\lambda_{\pi\sigma}} = \frac{\gamma_{\sigma\pi}}{\gamma_{\pi\sigma}} = \frac{N_{\pi}}{N_{\sigma}}, \quad (10)$$

where $N_{\pi}/N_{\sigma} \sim 1.3$ for MgB₂⁶. We use the values of $\lambda_{\sigma\sigma}$, $\lambda_{\pi\pi}$, $\lambda_{\sigma\pi}$ and $\lambda_{\pi\sigma}$ fixed to be 0.81, 0.285, 0.119 and 0.09, respectively in the following calculations^{4,9}. In this way, we determined the values of g for six films using the experimentally obtained T_c , as listed in Table II.

The equation for calculating H_{c2} is presented as follows,

$$(\lambda_0 + \lambda_i)[\ln t + U(x_+)] + (\lambda_0 - \lambda_i)[\ln t + U(x_-)] + 2w[\ln t + U(x_+)] = 0, \quad (11)$$

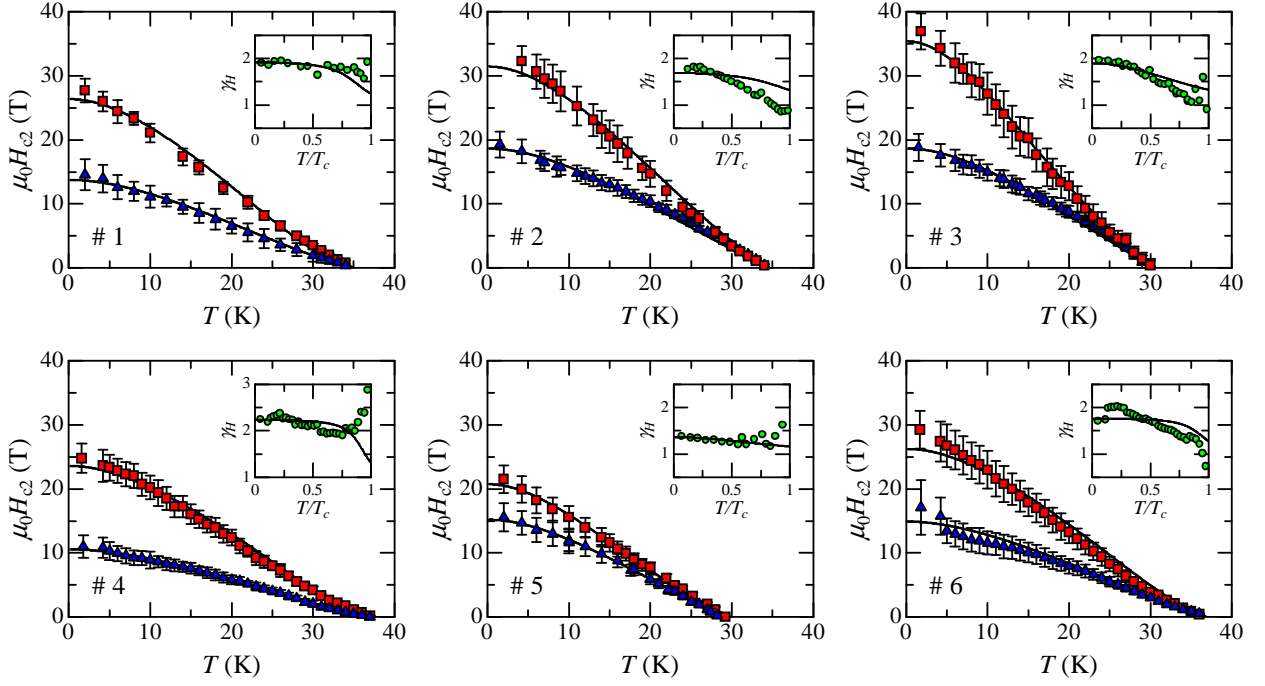


FIG. 4: $H_{c2}(T)$ curves of six films for the field direction $H \parallel ab$ -plane (square) and $H \parallel c$ -axis (triangle). Insets show temperature dependence of anisotropy parameter. Solid lines are calculation curves obtained from fitting the Gurevich theory.

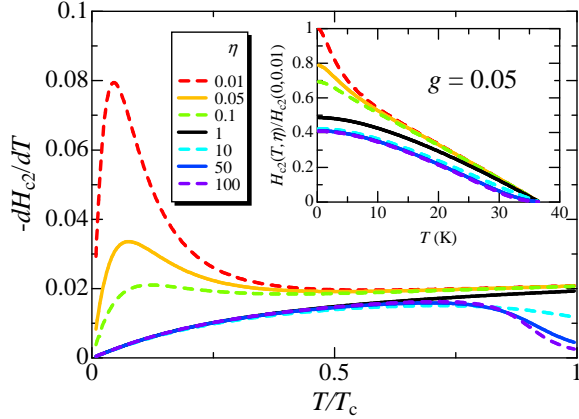


FIG. 5: Calculation curves of the Gurevich theory in $-dH_{c2}/dT$ vs. T/T_c plot under several values of anisotropy parameter η . A parameter g is fixed to be 0.05. Inset shows the calculated $H_{c2}(T)$ curves with the same parameters.

$$\lambda_i = [(\omega_- + \gamma_-)\lambda_- - 2\lambda_{\sigma\pi}\gamma_{\pi\sigma} - 2\lambda_{\pi\sigma}\gamma_{\sigma\pi}]/\Omega_0, \quad (12)$$

$$x_{\pm} = \hbar(\omega_{\pm} + \gamma_{\pm} \pm \Omega_0)/4\pi k_B T, \quad (13)$$

$$\Omega_0 = [(\omega_- + \gamma_-)^2 + 4\gamma_{\sigma\pi}\gamma_{\pi\sigma}]^{1/2}, \quad (14)$$

$$\omega_{\pm} = (D_{\sigma} \pm D_{\pi})\pi\mu_0 H/\phi_0, \quad (15)$$

where $t = T/T_{c0}$ and ϕ_0 is the flux quantum. D_{σ} and D_{π} are the intraband electron diffusivities in the σ and π bands, respectively. Since the π band has three-dimensional nature much more than the σ band, we assume isotropic diffusivity in the π band; $D_{\pi} = D_{\pi}^{(ab)} =$

$D_{\pi}^{(c)}$. In the σ band, on the other hand, anisotropic diffusivities along c -axis and ab -plane are introduced; $D_{\sigma} = D_{\sigma}^{(ab)}$ for $H \parallel c$ -axis, while $D_{\sigma} = \sqrt{D_{\sigma}^{(ab)} D_{\sigma}^{(c)}}$ for $H \parallel ab$ -plane. $D_{\sigma}^{(ab)}$ and $D_{\sigma}^{(c)}$ are the diffusivities along the ab -plane and the c -axis, respectively. The ratio $D_{\pi}/D_{\sigma} = \eta$ is defined as $\eta^{(ab)} = D_{\pi}/\sqrt{D_{\sigma}^{(ab)} D_{\sigma}^{(c)}}$ for $H \parallel ab$ -plane and $\eta^{(c)} = D_{\pi}/D_{\sigma}^{(ab)}$ for $H \parallel c$ -axis, respectively.

In the calculation, the absolute value of D_{σ} is roughly determined from the value of the $H_{c2}(0)$ of each field direction. On the other hand, the shape of the $H_{c2}(T)$ curve is mainly dependent on the parameter η . In the case of $\eta \ll 1$, the curve shows a steep increase at a low temperature near 0 K, while in the case of $\eta \gg 1$, the initial slope of the $H_{c2}(T)$ curve at T_c is suppressed as shown in the inset of Fig. 5. As $\eta = 1$, the $H_{c2}(T)$ curve is identical to the curve for a single-band superconductor. This situation is much clear in the differential plot, $-dH_{c2}/dT$ vs. T/T_c , as shown in Fig. 5. Therefore, in fitting the $H_{c2}(T)$ curves, we also referred the $-dH_{c2}/dT$ plot as shown in Fig. 6. It becomes quite valuable in these advanced analyses that we measured the magnetoresistance at a lot of temperatures for each film. In this way, we successfully obtain the calculation curves drawn by solid lines in Fig. 4 and Fig. 6, which show fairly good agreement with the experimental data points. Obtained parameters are listed in Table II.

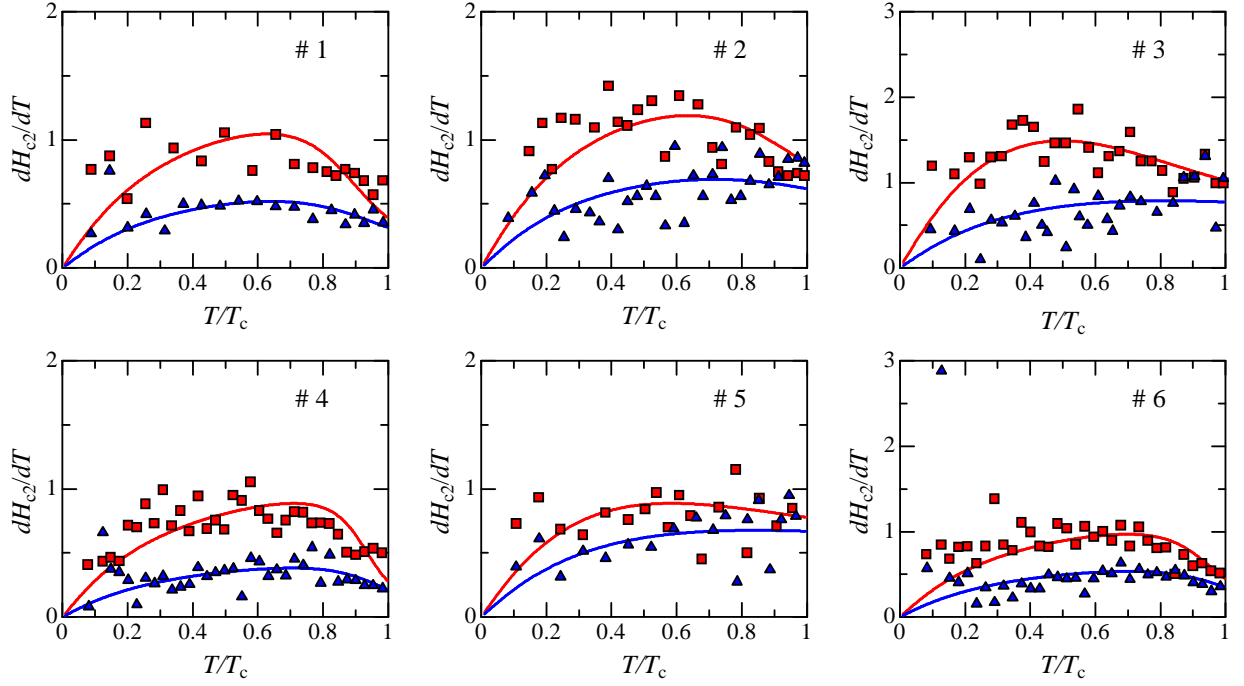


FIG. 6: dH_{c2}/dT vs. T plots of six films for the field direction $H \parallel ab$ -plane and $H \parallel c$ -axis. Solid lines are fitting curves by using the Gurevich theory with the same parameters as shown in Fig. 4.

TABLE II: Experimentally obtained values of some superconducting characteristics and the best fitting parameters of Gurevich calculation to the experimental data.

Film No.	experimental				fitting parameters					
	T_c (K)	$\mu_0 H_{c2}^{(ab)}(0)$ (T)	$\mu_0 H_{c2}^{(c)}(0)$ (T)	$\gamma_H(0)$	g	D_π (cm ² /s)	$D_\sigma^{(ab)}$ (cm ² /s)	$D_\sigma^{(c)}$ (cm ² /s)	$\eta^{(ab)}$	$\eta^{(c)}$
#1	35.1	29.1	15.3	1.91	0.077	20.88	1.48	0.38	27.7	14.1
#2	34.5	35.4	20.0	1.78	0.089	6.92	1.08	0.35	11.3	6.4
#3	30.4	38.6	19.5	1.98	0.197	3.85	0.70	0.11	13.8	5.5
#4	37.1	25.3	11.4	2.23	0.042	44.16	2.28	0.45	43.9	19.4
#5	28.8	23.0	16.3	1.41	0.265	4.20	0.65	0.19	12.1	6.5
#6	36.9	30.3	18.6	1.62	0.046	22.78	1.60	0.50	25.4	14.3

V. DISCUSSIONS

The results of the analysis as listed in Table II show the case of $\eta > 1$ for all MgB₂ films, which means the films have the cleaner π band than the σ band. This is confirmed also in the temperature dependence of γ_H as shown in the insets of Fig. 4, where γ_H decreases with increasing temperature, which is explained as the case of $\eta > 1$ in the Gurevich theory⁶. There are many reports of the disorder effect on H_{c2} by doping a nonmagnetic impurity to MgB₂ bulks and films. For example, the H_{c2} attains up to 60 T in the C substitution system⁸. On the contrary, our films were fabricated with no artificially doped impurity to get high grade films for device applications. Nevertheless, even such films show an enhancement of the H_{c2} , which suggests that the structural disorder is more dominant than the substitution of nonmagnetic impurity atoms. Several factors to cause

the structural disorder are deviation of the stoichiometry, mismatch of the lattice constants between MgB₂ and substrates (which causes a strain or distribution of the interatomic distance), randomness of in-plane orientation, degradation by the oxygen and water attack, and so on. It is quite natural that the structural disorder is equally introduced in both σ and π bands as well as in the interband scattering.

In order to make clear the disorder effect, we plot several parameters obtained from this study against the T_c as shown in Fig. 7. The experimental parameters, $\mu_0 H_{c2}^{(ab)}(0)$, $\mu_0 H_{c2}^{(c)}(0)$ and γ_H exhibit no T_c dependence as shown in the upper graph of Fig. 7. On the other hand, obtained parameters from the analysis, D_π , $D_\sigma^{(ab)}$ and $D_\sigma^{(c)}$ decrease clearly with decreasing T_c . $D_\sigma^{(ab)}$ and $D_\sigma^{(c)}$ show an exponential dependence on the T_c , as drawn by solid lines in the middle graph of Fig. 7. D_π de-

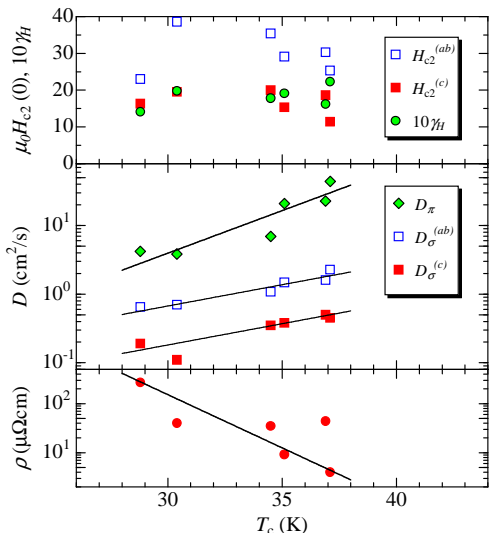


FIG. 7: Plots of several parameters obtained from this study against the T_c .

TABLE III: Several parameters fitting the Gurevich calculation to the experimental data of MgB_2 film #3 for different definitions of the H_{c2} .

#3	T_c (K)	g	D_π (cm^2/s)	$D_\sigma^{(ab)}$ (cm^2/s)	$D_\sigma^{(c)}$ (cm^2/s)
midpoint	30.4	0.197	3.85	0.70	0.11
90% R_n	31.3	0.168	4.05	0.72	0.14
99% R_n	32.4	0.137	6.19	0.68	0.20

creases much faster than $D_\sigma^{(ab)}$ and $D_\sigma^{(c)}$ with decreasing T_c . Also, $\eta^{(ab)}$ and $\eta^{(c)}$ show similar T_c dependence to the diffusivity, though they are not plotted in Fig. 7. As for the resistivity, ρ increases roughly with decreasing T_c as shown in the bottom graph of the figure. As a conclusion, T_c of the films is a good parameter to characterize the quality of the superconducting films even in two-band superconductors, as well as the residual resistivity, although there are many kinds of disorders on fabricating the MgB_2 films, such as non-stoichiometry, mismatch of the lattice constants between substrates and MgB_2 , atmosphere or degree of vacuum, degradation by oxygen or water, and so on.

Next, we discuss the definition of the H_{c2} . The H_{c2} defined as an onset of the magnetoresistive transition corresponds to a thermodynamical H_{c2} as usually argued in the theoretical treatments. However, the onset fields determined experimentally are less relevant due to defocus of the transition than the midpoint fields which are also coincident with the peak fields of the dR/dH plots as mentioned before. Therefore, the midpoint definition is much relevant from an experimental point of view in the systematic measurements and characterizations of various MgB_2 thin films fabricated by different methods²³. Nevertheless, it is worth to check how obtained parameters from the calculation fitting to the experimental data

are varied by the difference of the H_{c2} definitions. The magnetoresistance curves of the film #3 are rather sharp as shown in Fig. 3, so we obtained each $H_{c2}(T)$ determined from midpoint, 90% and 99% of the normal resistance (R_n). Although the T_c 's and the $H_{c2}(0)$'s are different according to the definition, the temperature dependence of the H_{c2} among three definitions is very similar to one another. Then, we also analyzed each data of the $H_{c2}(T)$ by fitting the Gurevich theory. Obtained parameters are listed in Table III. The parameters D_π , $D_\sigma^{(ab)}$ and $D_\sigma^{(c)}$ for 90% R_n are almost the same as those for the midpoint definition. On the other hand, D_π and $D_\sigma^{(c)}$ for 99% R_n are about 1.5 times larger than those for the others. However, when we plot these data to Fig. 7, we find that they are plotted near the solid line of the figure. they are almost plotted on the line drawn in Fig. 7. Accordingly, the conclusion of the cleaner π band is not changed by the difference of the H_{c2} definition.

Finally, we discuss briefly the transition width in the magnetoresistance measurements. There are no discussions on the transition width in the two-band superconductivity as far as we know. As shown in Fig. 3, the magnetoresistive transition becomes sharp at 26 K and broad below that temperature. If we interpret this behavior only in terms of the $H_{c2}(T)$ phase diagram, it may be related to them that the $H_{c2}(T)$ curve shows an upward curvature. When the superconductivity in the π band is destroyed by applied field, the normal core appears partly to form a vortex pinning center. The pinning mechanism of MgB_2 films has been speculatively discussed from the mixed-state transport measurements by Arcos and Kunchur²⁴. Although the transition width is often related to the local T_c variations due to some inhomogeneties, it may be attributed to two-band superconducting characteristics. Here, we point out that the transition width observed in some MgB_2 thin films does not show monotonic temperature dependence²⁵.

VI. CONCLUSION

We presented experimental results on the $H_{c2}(T)$ for $H \parallel ab$ -plane and $H \parallel c$ -axis of various as-grown MgB_2 thin films prepared by the molecular beam epitaxy, multiple-targets sputtering, and co-evaporation deposition apparatus. The results were well described by the Gurevich theory of dirty two-gap superconductivity. We extracted empirical parameters on the diffusivity of superconducting electrons for the σ and π bands from the $H_{c2}(T)$ calculation. All films were categorized to $D_\pi/D_\sigma > 1$, namely the cleaner π band case. We found that the parameters obtained from the analysis are strongly correlated to the T_c of the films. Accordingly, T_c is a good parameter for characterizing the quality of the films in a view of superconducting properties, as well as the residual resistivity.

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